Welcome to the College of Microscopy!

Food and beverages are not permitted in the auditorium, please enjoy them in the Atrium or Café.

Power receptacles are located between every other seat for your convenience.
Microanalysis of Particles

a

Microbeam Analysis Society

Hosted by the

College of Microscopy

Westmont, IL

April 20-23, 2009
Welcome!
Introduction
Conference Format

Mornings and immediately after lunch will be devoted to lectures here in the auditorium. During the morning coffee break please be sure to sign up for two of the workshops. They're repeated so don't worry…

For the afternoon workshops, please seek out an escort holding a sign with the session name you selected and follow them to the session.

Lunch will be brought in so that our schedule can remain on track. Posters are up for the duration, please examine them at your leisure, or during the afternoon coffee/poster sessions.
The Banquet tonight:

buses will take us to ANL and return us here.

Be prepared for the bus to leave at 4:30.

The buses will bring us back after our tour of the APS.

Tomorrow evening stick around or come back for an evening with EDAX.
Thank you to the Generosity of our Sponsors
Microanalysis of Particles: An Overview
What is a particle?

The definition depends on one's scientific specialization.

The size ranges used by atmospheric scientists cover the primary size ranges.

In ecology it might be defined as a small object.

In nanotechnology it is defined as a small object that behaves as a whole unit in terms of its transport and its properties.

Frac. Size range

PM$_{10}$ (thoracic frac. $\leq 10 \ \mu$m)

PM$_{2.5}$ (respirable frac. $\leq 2.5 \ \mu$m)

PM$_{1}$ $\leq 1 \ \mu$m

Ultrafine (UFP or UP) $\leq 0.1 \ \mu$m

PM$_{1}$ – PM$_{2.5}$ (coarse frac. $2.5 \ \mu$m – $10 \ \mu$m)
Particles are contaminants, flaws in emulsions or coatings, thin film inconsistencies, fabrication failures, mysterious powders or something else, particles are the root cause of many industrial, environmental, and national security concerns.
What is that particle made of?

Enter the realm of nanoanalysis!
Complementary Analytical Approach
Sample isolation and preparation the key to successful analysis
Some techniques require addiGonal, highly specialized sample preparaGon methods. For example, TEM/AEM require samples, even parGcles to be sub‐sampled in order to provide successful results. FIB‐SEM instruments provide very exciGng opportuniGes for sample preparaGon as well as even potenGally more incredible opportuniGes for in place milling and sample characterizaGon. New bench top equipment for sample preparaGon that is much more economiGcally achievable.
We’ll hear about the latest efforts to apply new "extreme" geometry corrections to the cadre of quantification schemes. Plus we’ll learn how to utilize edge simulation tools to test whether the information we measure matches what we think we know about it, or is that the other way around…
Generational comparison
5 kV ComposiGon of the catalyst particles identified by EDS.
We'll hear about efforts to compensate for the geometric effects, we'll see the latest efforts obtained, additional latest generations of electron microscopy equipment, learn about obtaining crystal structure information from particles around 10 micrometers in diameter, and learn about cool new bright source techniques at the APS.
Lou Ross extends his greetings and his apologies.

Qualitative EDS: Understanding the basics
Concepts to keep in mind

- **Electron Beam**
- **Secondary electrons** (in the nm range)
- **Backscattered electrons** (in the several 10s of nm range to 100s of nms or μm range)
- **Characteristic X-rays** (in the μm range)
- **Auger Electrons**
- **Continuum X-rays**
- **Fluorescent X-rays**
\[ \sqrt{E} = C_1 \times (Z - C_2) \]

\( C_1, C_2 \) are constants
Two more major factors which influence X-rays emitted from sample:

**X-ray Absorption (A):** *photoelectric effect*: ionization of inner shell electrons.

Described by **Beer’s Law**:

\[
\frac{\mu}{\rho} = \text{mass absorption coefficient}
\]

\[
I = I_0 \times e^{-\left(\frac{\mu}{\rho}\right) \cdot \rho t}
\]

= density of material

= thickness (exponential dependence)

Jumps in \(\frac{\mu}{\rho}\) at critical ionization energies in sample: **X-ray absorption edges**

**Secondary X-ray Fluorescence (F):** production of secondary X-rays from ionization from X-ray absorption:

Area of secondary fluorescence can be larger than electron interaction volume, since X-rays are more penetrating than electrons.

Can be produced by characteristic and continuum X-rays
Absorption is not constant for samples with geometry.
Analysis of a PbO particle 1 μm in diameter.
The interaction volumes producing the various electrons and X radiation are different for each atom in the sample.

- Electron beam energy determines the interaction volume of sample
- Characteristic X-rays are produced by ionization of atoms in sample
- Energy of characteristic X-rays used for qualitative analysis
- Intensity of characteristic X-rays affected by operating conditions and sample characteristics (**matrix effects**)
- Intensity of characteristic x-rays affected by atomic number (**Z**), absorption (**A**), secondary fluorescence (**F**)
- Intensities of X-ray lines can be used for quantitative analysis **provided one has similar standard reference materials**
- For particles, geometry and size have huge influence
An electron imaging example of the Basics
Thank you for your participation, thank you to the speakers, and thank you to our sponsors and host!